इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”
Mazdoor Kisan Shakti Sangathan
“The Right to Information, The Right to Live”

“पुराने को छोड नये के तरफ”
Jawaharlal Nehru
“Step Out From the Old to the New”

IS 2766-1 (1968): Methods of Chemical Analysis of Primary Nickel - Part I [MTD 9: Lead, Zinc, Cadmium, Tin, Antimony and their Alloys]

“ज्ञान से एक नये भारत का निर्माण”
Satyanarayan Gangaram Pitroda
“Invent a New India Using Knowledge”

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”
Bhartṛhari—Nītiśatakam
“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE

BRUTUM FULMEN
IGNORANTIA JURIS NON EXCUSAT
PROTECTED BY COPYRIGHT

IS : 2766 (Part I) - 1968
(Reaffirmed 2000)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF PRIMARY NICKEL,
PART I

UDC 669·24 : 543

© *Copyright* 1968

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

Price Rs 6·00 Gr 5

April 1968

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF PRIMARY NICKEL, PART I

Methods of Chemical Analysis Sectional Committee, SMDC 2

Chairman	*Representing*
Dr G. V. L. N. Murty	The Tata Iron & Steel Co Ltd, Jamshedpur
Members	
Shri H. P. Samanta (*Alternate* to Dr G. V. L. N. Murty)	
Shri K. L. Banerjee	National Test House, Calcutta
Shri N. C. Sen Gupta (*Alternate*)	
Dr A. S. Bhaduri	Ministry of Railways
Shri P. Dutta (*Alternate*)	
Shri J. Bhattacharjee	Ministry of Defence (DGI)
Dr H. P. Bhattacharya	National Metallurgical Laboratory (CSIR), Jamshedpur
Dr U. N. Bhrany	Indian Iron & Steel Co Ltd, Burnpur
Shri A. C. Mukherjee (*Alternate*)	
Dr B. N. Chakaravarthi	Bhilai Steel Plant, Bhilai
Dr S. R. Iyengar (*Alternate*)	
Shri D. K. Chakravarty	Directorate General of Ordnance Factories (Ministry of Defence)
Deputy General Manager, Ordnance Factory, Kanpur (*Alternate*)	
Shri D. Sen (*Alternate*)	
Shri A. K. Chatterjee	The Binani Metal Works Limited, Calcutta
Dr A. N. Chowdhury	Geological Survey of India, Calcutta
Shri B. N. Bhattacharya (*Alternate*)	
Shri O. P. Dey	Naval Chemical and Metallurgical Laboratory [Ministry of Defence (R&D)]
Shri A. D. Gupta (*Alternate*)	
Dr R. K. Dutta	Durgapur Steel Plant, Durgapur
Shri A. C. Bhowmik (*Alternate*)	
Shri C. R. Goil	Ferro Alloys Corporation Ltd, Shreeramnagar
Shri G. V. Halwe	Directorate General of Supplies & Disposals (Inspection Wing), New Delhi
Shri S. N. Das Gupta (*Alternate*)	
Shri S. S. Honavar	Italab Private Ltd, Bombay
Shri J. P. Patel (*Alternate*)	
Shri J. C. Dey (*Alternate*)	

(*Continued on page* 2)

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

(*Continued from page* 1)

Members	*Representing*
DR N. JAYARAMAN	Essen & Co, Bangalore
SHRI M. R. G. SHARMA (*Alternate*)	
DR M. K. JOSHI	Defence Metallurgical Research Laboratory, Hyderabad
SHRI S. L. N. ACHARYULU (*Alternate*)	
DR S. KUMAR	Central Glass and Ceramic Research Institute (CSIR), Calcutta
SHRI B. K. AGARWAL (*Alternate*)	
SHRI N. C. MITRA	India Government Mint (Ministry of Finance), Calcutta
SHRI M. M. MOUDGILL	Indian Non-Ferrous Metals Manufacturers' Association, Calcutta
SHRI N. MAZUMDAR (*Alternate*)	
SHRI K. P. ROY CHOWDHURY	The Eyre Smelting Private Limited, Calcutta
SHRI A. SANGAMESWARA RAO	Indian Metals and Ferro Alloys Ltd, Koraput
DR P. R. SUBBARAMAN	National Chemical Laboratory (CSIR), Poona
DR CH. VENKATESWARLU	Bhabha Atomic Research Centre, Bombay
SHRI M. R. VERMA	National Physical Laboratory (CSIR), New Delhi
DR A. K. CHATTERJEE, Director (Struc & Met)	Director General, ISI (*Ex-officio Member*)

Secretary

SHRI S. L. BALI

Deputy Director (Met), ISI

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF PRIMARY NICKEL, PART I

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 5 February 1968, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 The purpose of this standard is to recommend standard methods for analysis of primary nickel covered by IS : 2782-1964*. Taking into consideration the different composition ranges of primary nickel as specified in Grades 1 and 2 of IS : 2782-1964*, complete analysis of primary nickel has been covered in parts. This part covers the analysis of nickel, cobalt and copper (Grades 1 and 2), sulphur, carbon and iron (Grade 2). The analysis of trace impurities, namely, sulphur, carbon, iron (Grade 1), antimony, bismuth and lead (Grades 1 and 2) will be covered in subsequent parts.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and the practices prevailing in different countries, in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the following publications:

Book of ASTM methods for chemical analysis of metals. Part 32. 1967. American Society for Testing and Materials.

FURMAN (N H), ED. and SCOTT (W W). Standard methods of chemical analysis. 1962. Ed 5. D. Van Nostrand Company, Inc, New York.

0.4 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

---

*Specification for primary nickel.

†Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard covers methods of chemical analysis of nickel, cobalt and copper (Grades 1 and 2), sulphur, carbon and iron (Grade 2) of primary nickel covered by IS : 2782-1964*.

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with the procedure laid down for the purpose of chemical analysis in IS : 2782-1964*.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. GENERAL

4.1 Use of Filter Papers — In some of the methods prescribed in this standard, relative numbers of Whatman filter papers which are commonly used have been specified. However, any other suitable brand of filter papers of corresponding porosity may also be used.

5. DETERMINATION OF NICKEL BY THE GRAVIMETRIC METHOD

5.1 Outline of the Method — The solution of the sample is neutralized with ammonia. From the solution, nickel is precipitated as nickel dimethylglyoxime in presence of ammonium citrate, filtered and weighed.

5.2 Reagents

5.2.1 *Dilute Nitric Acid* — 1 : 1 (*v/v*).

5.2.2 *Concentrated Sulphuric Acid* — sp gr 1·84 (conforming to IS : 266-1961‡).

5.2.3 *Dilute Hydrochloric Acid* — 5 : 95 (*v/v*).

5.2.4 *Citric (Tartaric) Acid* — 250 g/l.

5.2.5 *Concentrated Ammonium Hydroxide* — 20 percent.

---

*Specification for primary nickel.

†Specification for water, distilled quality (*revised*).

‡Specification for sulphuric acid (*revised*).

5.2.6 *Dimethylglyoxime Solution* — 1 percent (*w/v*) in rectified spirit. Filter the solution before use.

5.3 Procedure

5.3.1 Transfer 0·1 to 0·2 g of an accurately weighed sample in 600-ml beaker. Add 20 ml of dilute nitric acid and warm gently until reaction ceases. Add 5 to 7 ml of concentrated sulphuric acid and evaporate to white fumes. Allow the beaker to cool and then add 100 ml of water when nickel salts dissolve. Filter off the residue, if any, and then wash with dilute hydrochloric acid and finally with hot water.

5.3.2 Add 20 ml of citric (tartaric) acid and then add ammonium hydroxide until a slight odour is detected. Dilute with water to 400 ml and then heat the soluion to 60° to 80°C.

5.3.3 Add 40 ml of the dimethylglyoxime solution for each 0·1 g of the nickel present. Add 5 to 10 ml in excess. Render the solution slightly alkaline with concentrated ammonium hydroxide and digest at 60°C for about 30 minutes. Stir vigorously and allow to cool to room temperature slowly while stirring occasionally. Filter the precipitate on a weighed sintered crucible (G-3, 30 to 50 ml capacity) and wash it with warm water. Keep the filtrate overnight to make sure that nickel has been completely precipitated.

5.3.4 Dry the sintered glass crucible along with the precipitate at 130°C to constant weight. Cool in a desiccator and weigh as nickel dimethylglyoxime.

5.4 Calculation

$$\text{Nickel, percent} = \frac{A \times 20{\cdot}32}{B}$$

where

A = weight in g of nickel dimethylglyoxime, and

B = weight in g of the sample taken.

6. DETERMINATION OF COBALT BY THE NITROSO-R-SALT (PHOTOMETRIC) METHOD

6.1 Outline of the Method — After the removal of interfering elements like copper and iron, the hot solution of the sample buffered with sodium acetate is complexed with nitroso-R-salt and photometric measurement is made of an orange coloured complex at approximately 520 millimicrons (mμm).

6.2 Reagents

6.2.1 *Aqua Regia* — Mix concentrated hydrochloric acid (sp gr 1·16) and concentrated nitric acid (sp gr 1·42) in the ratio of 3 : 1 (*v/v*).

6.2.2 *Dilute Sulphuric Acid* — 1 : 1 (*v/v*).

6.2.3 *Hydrogen Sulphide* — gas.

6.2.4 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*).

6.2.5 *Nitroso-R-Salt Solution* — 1 percent (*w/v*). Dissolve 0·10 g of nitroso-R-salt in water, filter and dilute to 100 ml. Do not keep the solution for more than a week.

6.2.6 *Sodium Acetate* — solid.

6.2.7 *Concentrated Hydrochloric Acid* — sp gr 1·16 (conforming to IS : 265-1962*).

6.2.8 *Standard Cobalt Solution* (*1* ml = *0·1* mg *of* Co) — Dissolve 0·67 g of pure cobalt ammonium sulphate (AR) [$CoSO_4\,(NH_4)_2SO_4,6H_2O$] in water and dilute to 1 litre.

6.3 Procedure

6.3.1 Dissolve 0·5 g of the sample in a mixture of 5 ml of aqua regia in a 150-ml beaker and evaporate to fumes with 3 to 5 ml of dilute sulphuric acid. Cool and dilute with about 200 ml of water. Warm to dissolve the salts.

6.3.2 Pass hydrogen sulphide through the hot solution and continue passing the gas till the solution cools down. Add a small amount of paper pulp and filter the sulphides. Transfer the solution to a 1-litre volumetric flask and make up the volume to the mark.

6.3.3 Take a suitable aliquot from the solution so that it contains between 0·005 and 0·1 mg of cobalt. Evaporate almost to dryness, dissolve the residue in 10 ml of water containing 0·5 ml each of dilute hydrochloride acid and nitroso-R-salt solution. Add 2 g of sodium acetate. The *p*H of the solution at this point should be 5·5. Boil for a minute, cool and add 1·5 ml of concentrated hydrochloric acid. Boil further for a minute, cool and dilute to 50 ml in a volumetric flask.

6.3.4 Transfer a suitable portion of the solution prepared under **6.3.3** to an absorption cell and take the photometric reading using light filter at 520 millimicrons (m μ m).

6.3.5 Take a series of standard solutions of cobalt and a reagent blank. Using the same quantities of reagents carry through all stages as described under **6.3.3** and **6.3.4,** and record the photometric readings of all the standard solutions along with the blank. Construct a calibration curve by plotting the photometric readings of standard solutions against the quantities of cobalt.

---

*Specification for hydrochloric acid (*revised*).

6.3.6 Convert the photometric reading of the sample to milligrams of cobalt by means of the calibration curve and calculate as under **6.4.**

6.4 Calculation

$$\text{Cobalt, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of cobalt found in the aliquot of the solution, and

B = weight in g of the sample represented by the aliquot taken.

7. DETERMINATION OF TOTAL CARBON BY THE DIRECT COMBUSTION (GRAVIMETRIC) METHOD

7.1 Outline of the Method — The sample is heated to combustion at a high temperature (1 200° to 1 300°C) in a stream of pure oxygen and the carbon is converted to carbon dioxide. The carbon dioxide, carried by oxygen, is passed through and absorbed by soda asbestos in a tared bulb. The increase in weight of the bulb is proportional to the quantity of carbon dioxide formed.

7.2 Reagents

7.2.1 *Oxygen* — 99·5 percent purity, free from carbonaceous matter.

7.2.2 *Anhydrous Magnesium Perchlorate* — $Mg(ClO_4)_2$.

7.2.3 *Flux* — lead dioxide, analytical reagent grade.

7.2.4 *Manganese Dioxide* — If a suitable chemical active grade is not available, it may be prepared as follows:

a) To prepare about 50 g of manganese dioxide, dissolve in a 4-litre beaker 200 g of manganous sulphate ($MnSO_4,4H_2O$) in 2·5 litres of water. After making this solution clearly ammoniacal, add 1 litre of ammonium persulphate solution (225 g/l) freshly prepared, bring to boiling point. Continue boiling for 10 minutes and add ammonia solution as frequently as is necessary to maintain the solution ammoniacal. Allow the precipitate to settle.

b) If the supernatant liquid is not clear or if the precipitate does not settle quickly, add 50 to 100 ml of ammonium persulphate solution and boil again for 10 minutes keeping the solution constantly ammoniacal.

c) When precipitation appears to be complete allow the manganese dioxide to settle completely, carefully siphon off the supernatant liquid and wash the precipitate by decantation with 3 or 4 litres of warm water in portions of 500 to 600 ml. Stir the manganese dioxide well in the water and allow to settle after each washing and before decantation. Finally wash twice in the same way with very dilute sulphuric acid.

d) In the meantime, prepare a 15-cm diameter funnel fitted with a 5-cm filter disc filled with a thin layer of purified asbestos pulp (in place of the latter, it is also permitted to use a porcelain funnel of the Buchner type).

e) After the last washing, transfer the manganese dioxide into the filter and wash with warm water until it is free from sulphate ions. Then place this on a porcelain dish and dry it in an oven at 105°C.

f) Grind the manganese dioxide in a mortar so that it will pass through a sieve with apertures of 0·8 mm and dry again completely at 105°C.

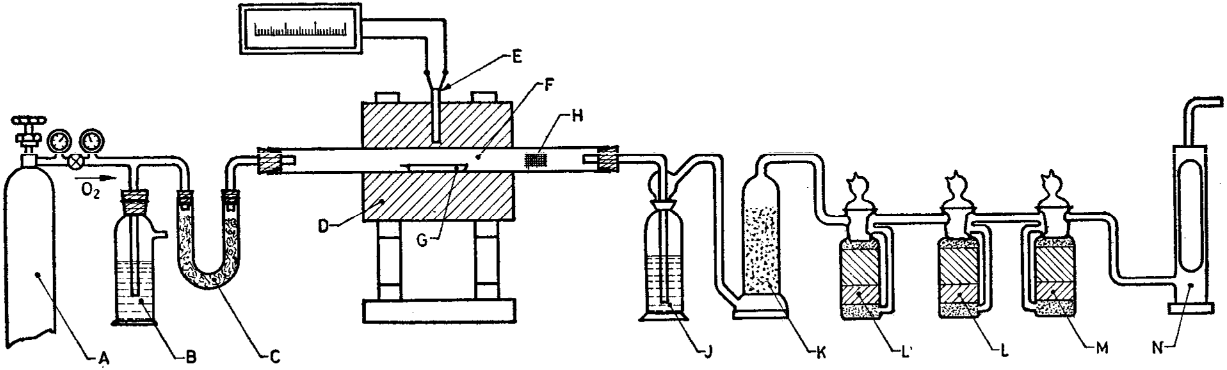
7.2.5 *Soda Asbestos* — asbestos impregnated with sodium hydroxide, in granules of about 2 mm diameter. Avoid contact with air.

7.3 Apparatus — It consists of a source of oxygen and the unit for purifying it, the furnace with the combustion tube, the purification train and the carbon dioxide absorption system. These different parts, which are joined together with connecting tubes forming an air-tight seal, are shown in Fig. 1.

7.4 Procedure

7.4.1 After having verified the pressure tightness of the apparatus and the efficiency of the oxygen purification and having reached the required temperature (1 200° to 1 300°C) in the combustion zone of the furnace, allow oxygen to pass through the apparatus for 10 to 15 minutes at the rate of 300 to 500 ml/min of gas depending on the diameter of the tube used. Then disconnect the absorption bulbs and weigh them at the ambient temperature and replace them in position.

7.4.2 Place 1 g of an accurately weighed sample in the form of very short chips having a thickness of a few tenths of a millimetre, in a boat. If necessary, add to the sample 1 to 2 g of flux (**7.2.3**).



A Source of oxygen (**7.2.1**) with pressure regulating valve.
B Mercury valve.
C Unit for drying and purifying the oxygen, containing anhydrous magnesium perchlorate (**7.2.2**) and soda asbestos (**7.2.5**), separated by glass wool U tubes, 25 mm diameter and 100 mm height approximately) connected by tubing.
D Wire-wound or resistor-rod furnace, made of carborundum or metal and capable of a temperature for combustion up to 1 300°C.
E Thermocouple for measuring the temperature. The tip of the thermocouple protected by a sheath, is placed near the external surface of the combustion tube. The relation between the internal tube temperature and the pyrometer reading should be established.
F Combustion tube made of refractory material which is not porous at the test temperature, internal diameter 20 to 30 mm and at least 650 mm long so that the ends of the tube remain cold during combustion.
G Boat made of refractory material (length 80 to 100 mm, width 12 to 14 mm and depth 8 to 9 mm), calcined in a steam of oxygen at the temperature of use.
H Plug of calcined asbestos wool or quartz wool to filter the gas.
J Washing bulb containing manganese dioxide (*see* **7.2.4**).
K Drying bulb containing anhydrous magnesium perchlorate (*see* **7.2.2**).
L Weighed absorption bulbs for absorbing the carbon dioxide, containing soda asbestos (**7.2.5**) and a certain amount of anhydrous magnesium perchlorate (**7.2.2**) to trap the water which forms during absorption of the carbon dioxide. The total weight of each absorption bulb ready for use (*see* Fig. 2) should not exceed 100 g.
M Unweighed absorption bulb facing the opposite way to *L*.
N Flowmeter.

FIG. 1 COMBUSTION APPARATUS FOR DETERMINATION OF CARBON BY GRAVIMETRIC METHOD

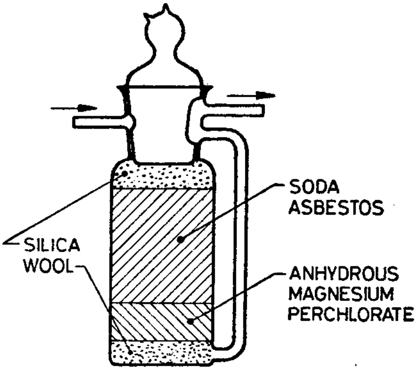


FIG. 2 ABSORPTION BULB

7.4.3 Open the combustion tube at the end where the oxygen enters, and using a rigid nickel wire place the boat and the sample in the middle of the heated zone of the tube. Quickly close the tube and after about 1 minute pass through it a stream of oxygen at the rate of 300 to 500 ml/min depending on the diameter of the tube used. After about 1 minute, when the combustion has ended, continue to pass oxygen through for another 15 to 20 minutes so that the carbon dioxide is completely removed from the combustion tube and the purifying bulbs.

7.4.4 Cut off the stream of oxygen, close the taps of the weighed absorption bulbs and remove the porcelain boat from the combustion tube. It is necessary to verify that the combustion has been efficiently completed by examining the fused weight in the boat removed from the furnace.

7.4.5 Disconnect the absorption bulbs and weigh them at ambient temperature. The increase in weight represents the carbon dioxide absorbed.

7.4.6 Carry out the blank test by the combustion of the boat in the presence of the quantity of flux used in the analysis, and carefully determine the weight of the absorption bulb.

7.5 Calculation

$$\text{Carbon, percent} = \frac{A - B}{C} \times 27{\cdot}3$$

where

A = weight in g of carbon dioxide obtained under **7.4.5**,

B = weight in g obtained from the blank, and

C = weight in g of the sample taken.

8. DETERMINATION OF IRON BY THE DICHROMATE (VOLUMETRIC) METHOD

8.1 Outline of the Method — Iron is precipitated as hydroxide from the solution of the sample and dissolved in hydrochloric acid. To the boiling solution, stannous chloride is added in slight excess to reduce iron. The unused stannous chloride is destroyed by mercuric chloride and the reduced iron titrated with standard dichromate solution using sodium diphenylamine sulphonate as indicator.

8.2 Reagents

8.2.1 *Concentrated Nitric Acid* — sp gr 1·42 (conforming to IS : 264-1950*).

8.2.2 *Concentrated Ammonium Hydroxide* — 20 percent.

8.2.3 *Dilute Ammonium Hydroxide* — 5 : 95 (*v/v*).

8.2.4 *Dilute Hydrochloric Acid* — 1 : 1, 1 : 3 and 5 : 95 (*v/v*).

8.2.5 *Stannous Chloride Solution* ($SnCl_2 2,H_2O$) — Dissolve by heating 60 g of pure stannous chloride in a mixture of 400 ml of concentrated hydrochloric acid and 600 ml of water until the solution is complete. Cool, add a few pieces of granulated tin and preserve the solution in an air-tight amber-coloured bottle to prevent oxidation.

8.2.6 *Mercuric Chloride Solution* — Prepare a saturated solution of mercuric chloride in water.

8.2.7 *Sulphuric-Phosphoric Acid Mixture* — Add slowly 150 ml of concentrated sulphuric acid (sp gr 1·84) to 700 ml of water with continuous stirring. Add to this 150 ml of syrupy phosphoric acid (85 percent). Destroy any oxidizable impurities by adding potassium permanganate solution (0·1 N), drop by drop, until the pink colour of permanganate persists, and cool the solution.

8.2.8 *Sodium Diphenylamine Sulphonate Indicator Solution* — Dissolve 0·32 g of barium diphneylamine sulphonate in 100 ml of hot water. Add 0·5 g of sodium sulphate, stir, allow to settle, and filter off the precipitate of barium sulphate. Store in a dark-coloured bottle.

8.2.9 *Standard Potassium Dichromate Solution* — 0·02 N. Dry pure potassium dichromate (AR) in an oven at 105° to 110°C for one and a half hours. Cool in a desiccator and transfer exactly 0·980 6 g to a

---

*Specification for nitric acid.

1-litre volumetric flask carefully through a funnel using water at room temperature. Wash the funnel thoroughly and dissolve the salt by shaking until the solution is complete. Make up to the mark and mix well.

8.3 Procedure

8.3.1 Dissolve 5 g of an accurately weighed sample in a 600-ml beaker in 35 ml of concentrated nitric acid. Heat the solution gently until action ceases. When solution is clear, boil it until brown fumes are no longer evolved, and dilute to 100 ml. Make the solution strongly ammoniacal, adding sufficient amount of concentrated ammonium hydroxide and boil for 1 minute. Filter the precipitate through filter paper No. 41 and wash with dilute ammonium hydroxide. Dissolve the precipitate with hot dilute hydrochloric acid (1 : 1) and reprecipitate iron and filter it. Dissolve the precipitate in 40 ml of hot dilute hydrochloric acid (1 : 3) receiving the solution in a 500-ml conical flask and washing the paper with hot dilute hydrochloric acid (5 : 95).

8.3.2 Heat the solution to boiling and, to the boiling solution, add stannous chloride solution, drop by drop, with continuous stirring until the solution just becomes colourless. Add 2 to 3 drops excess of stannous chloride solution and wash down the sides with a little water. Cool the flask rapidly under running tap water until the contents have cooled down to room temperature.

8.3.3 Add excess of mercuric chloride solution (about 5 ml) and shake the flask. At this stage, a silky white precipitate appears. If it fails to appear or if a blackish precipitate appears, reject the test. If the precipitable is too much, add further 5 ml of mercuric chloride solution. Keep for about 2 to 5 minutes and add 15 ml of sulphuric-phosphoric acid mixture, and dilute to about 250 ml, washing down the sides of the flask. Add 3 to 4 drops of sodium diphenylamine sulphonate indicator solution and titrate slowly with standard potassium dichromate solution stirring continuously until the green colour begins to darken. Continue the titration slowly till a drop of the dichromate solution produces a reddish violet colouration.

8.3.4 Carry out a blank determination following the same procedure and using the same amounts of all reagents, but without the sample.

8.4 Calculation

$$\text{Iron, percent} = \frac{(A - B)\,C \times 5{\cdot}585}{D}$$

where

A = volume in ml of the standard dichromate solution required for the sample,

B = volume in ml of the standard dichromate solution required for the blank,

C = normality of the standard dichromate solution, and

D = weight in g of the sample taken.

9. DETERMINATION OF SULPHUR BY THE GRAVIMETRIC METHOD

9.1 Outline of the Method — Sulphur in the sample is oxidized to sulphate and then precipitated as barium sulphate.

9.2 Reagents

9.2.1 *Sodium Chlorate* — solid.

9.2.2 *Concentrated Nitric Acid* — *See* **8.2.1**.

9.2.3 *Concentrated Hydrochloric Acid* — *See* **6.2.7**.

9.2.4 *Hydroxylamine Hydrochloride* — solid.

9.2.5 *Barium Chloride Solution* — 20 percent (*w/v*).

9.2.6 *Barium Chloride Wash Solution* — 0·2 percent (*w/v*). Add 10 ml of the solution (prepared under **9.2.5**) to 10 ml of concentrated hydrochloric acid (*see* **6.2.7**) and dilute to 1 litre.

9.3 Procedure

9.3.1 Transfer 10 g of an accurately weighed sample to a 80-ml beaker. Add 2 to 3 g of sodium chlorate and then 100 ml of concentrated nitric acid when vigorous reaction starts. When reaction subsides, warm the solution till reaction ceases and evaporate the solution to a small volume till it is syrupy. Allow the solution to cool and then add 50 ml of concentrated hydrochloric acid and evaporate the solution to dryness. Bake the residue on a hot plate until the salts assume a uniform yellow colour. Repeat the baking process by addition of 50 ml of concentrated hydrochloric acid.

9.3.2 Cool the baked mass and then moisten with 7 ml of concentrated hydrochloric acid. Heat the mixture at 60° to 70°C until any iron oxides have been converted to chlorides guarding against any appreciable loss of concentrated hydrochloric acid by evaporation. Add 150 ml of water and boil the solution until the salts have been dissolved. Filter the solution through a filter paper in a beaker and wash the filter paper with hot water.

9.3.3 Dilute the filtrate and washings to 350 ml and heat to boiling. Add 25 ml of barium chloride solution with constant stirring and allow the precipitate to settle on a water-bath.

9.3.4 Filter and collect the precipitate on to a fine filter, containing a small quantity of paper pulp, and use a jet of cold washing solution for washing of the residue from the beaker. Wash the precipitate on the filter 3 or 4 times with the washing solution, and then with warm water until the chlorine ions are completely absent from the washings (test reaction with silver nitrate).

9.3.5 Place the filter with the precipitate in a weighed platinum or porcelain crucible and dry. Carefully ignite and heat at 600° to 700°C for 30 minutes. Cool the crucible with the residue in a desiccator and then weigh.

9.3.6 Make a blank determination following the same procedure and using the same amounts of all reagents.

9.4 Calculation

$$\text{Sulphur, percent} = \frac{(A - B) \times 13{\cdot}74}{C}$$

where

A = weight in g of barium sulphate obtained in **9.3.5**,

B = weight in g of barium sulphate in the blank, and

C = weight in g of the sample taken.

10. DETERMINATION OF COPPER BY THE PHOTOMETRIC METHOD

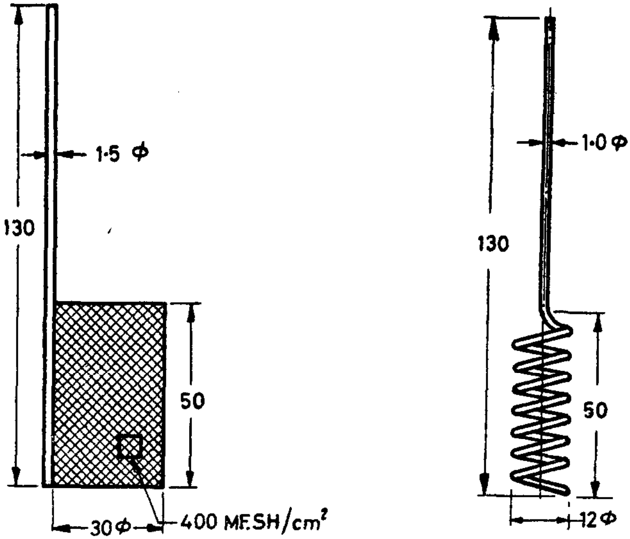
10.1 Outline of the Method — The sample is dissolved in nitric acid and sulphuric acid. Copper is separated by electrolysis. The copper deposit is dissolved in nitric acid and then determined photometrically.

10.2 Apparatus

10.2.1 *Electrodes* — The following platinum electrodes (*see* Fig. 3) are recommended, but strict adherence to the shape and size of the electrodes is not essential. For agitation of electrolyte in order to decrease the time of deposition, one of the types of rotating forms of electrodes, generally available, may be employed.

10.2.2 *Cathode* — It may be formed either from plain or from perforated sheet or from wire gauze.

10.2.2.1 Gauze cathodes preferably made from gauze containing 400 mesh/cm² should be used. The wire used for making gauze should be approximately 0·20 mm in diameter. Cathodes should be stiffened by doubling the gauze for about three millimetres on the top and the bottom or by reinforcing the gauze at the top and bottom with platinum ring or band.



3A Cylindrical Platinum Cathode 3B Spiral Platinum Anode

All dimensions in millimetres.

FIG. 3 CYLINDRICAL PLATINUM CATHODE AND SPIRAL PLATINUM ANODE

10.2.2.2 The diameter of the cylinder should be approximately 30 mm and the height 50 mm. The stem should be made from platinum alloy wire, such as platinum-iridium, platinum-rhodium or platinum-ruthenium having diameter of approximately 1·5 mm. It should be flattened and welded throughout the entire length of the gauze. The overall height of the cathode should be approximately 130 mm.

10.2.3 *Anode* — When the amount of lead in the sample is less than four milligrams, a spiral anode should be used. It should be made from 1·0 mm or larger platinum wire formed into spiral of seven turns with a height of approximately 50 mm and diameter of 12 mm, the overall height being 130 mm.

10.2.3.1 When the amount of lead in the sample is more than four milligrams, the gauze anode should be used. It should be made of the same material and of the same general design as platinum gauze cathode mentioned under **10.2.2.** It should be approximately 12 mm in diameter and 50 mm in height, the overall height being 130 mm.

10.3 Reagents

10.3.1 *Concentrated Nitric Acid — See* **8.2.1.**

10.3.2 *Dilute Sulphuric Acid* — 1 : 1 (*v*/*v*).

10.3.3 *Dilute Hydrochloric Acid* — 1 : 99 (*v*/*v*).

10.3.4 *Dilute Nitric Acid* — 1 : 3 (*v*/*v*).

10.3.5 *Concentrated Ammonium Hydroxide* — 20 percent.

10.3.6 *Citric Acid Solution* — 20 percent (*w*/*v*).

10.3.7 *Sodium Diethyl-Dithiocarbamate Solution* — 0·1 percent (*w*/*v*).

10.3.8 *Carbon Tetrachloride*

10.3.9 *Sodium Sulphate* — solid.

10.3.10 *Standard Copper Solution* — Dissolve 0·2 g of electrolytic copper in a minimum volume of dilute nitric acid (1 : 1). Dilute, neutralize with ammonia, reacidify with concentrated sulphuric acid and dilute to 1 litre.

10.4 Procedure

10.4.1 Dissolve 5 g of an accurately weighed sample at a low temperature in 35 ml of concentrated nitric acid in a 250-ml beaker. To this add 50 ml of water and 5 ml of dilute sulphuric acid and heat till the solution is clear.

10.4.2 Dilute the solution to 125 ml and add one drop of dilute hydrochloric acid. Electrolyze till deposition of copper is complete using platinum gauze electrodes at a current density of 0·3 to 0·4 A/dm^2. Lower the electrolytic beaker quickly, with the current still on. Rinse the cathode with water. Turn off the current quickly, detach the cathode and rinse it in a beaker of water.

10.4.3 Dissolve the deposited copper from the cathode by means of minimum quantity of hot dilute nitric acid. Cool and add sufficient ammonium hydroxide to make the solution alkaline and 1 ml in excess and shake volume into a 100-ml graduated flask with distilled water.

10.4.4 Pipette suitable aliquot (depending on the copper centent of the solution) into a 250-ml separating funnel. Add 10 ml of citric acid solution. Make distinctly ammoniacal. Add 10 ml of sodium diethyldithiocarbamate solution, mix well and allow to stand for 15 minutes. Extract successively with 25 ml, 15 ml and 10 ml of carbon tetrachloride. Combine carbon tetrachloride extracts and make up to 50 ml in a graduated flask. Add a little anhydrous sodium sulphate to clear the solution.

10.4.5 Transfer a suitable aliquot to an absorption cell and adjust the photometer to the initial setting. Take the photometric reading using a light filter at appproximately 435 millimicrons (mμm).

10.4.6 *Calibration Curve* — Take a series of standard copper solutions and a reagent blank. Using same quantities of all reagents, carry through all the stages as described under **10.4.4** and **10.4.5** and record the photometric readings of the standard solutions along with the blank. Draw a calibration curve by plotting the photometric readings of standard solutions against milligrams of copper per hundred ml of the solution.

10.5 Calculation — Convert the photometric readings of the sample to milligrams of copper by means of calibration curve, and calculate the percentage of copper as follows:

$$\text{Copper, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of copper found in the aliquot solution, and

B = weight in g of the sample represented by the aliquot taken.

NOTE — Since the carbon tetrachloride extract of copper complex is readily bleached by exposure to strong light, extraction should, therefore, be carried out in artificial light or weak daylight.

INDIAN STANDARDS

ON

Methods of Chemical Analysis

IS:		Rs
228-1959	Methods of chemical analysis of pig iron, cast iron and plain carbon and low-alloy steels (*revised*)	6·50
403-1964	Methods of chemical analysis of lead and antimonial lead (*revised*)	5·50
406-1964	Methods of chemical analysis of slab zinc (spelter) (*revised*)	3·50
440-1964	Methods of chemical analysis of copper (*revised*)	5·50
504-1963	Methods of chemical analysis of aluminium and its alloys (*revised*)	7·00
964-1956	Methods for chemical analysis of silver solder	1·50
998-1959	Methods of chemical analysis of soft solder	3·00
999-1959	Methods of chemical analysis of brazing solder	3·00
1047-1965	Methods of chemical analysis of antimony (*revised*)	5·00
1335-1959	Methods for the direct determination of alumina in refractory materials (*tentative*)	2·50
1338-1959	Certified samples for metallurgical analysis	1·00
1345-1960	Methods of chemical analysis of printing metals	3·50
1409-1959	Methods of chemical analysis of antifriction bearing alloys	4·00
1473-1960	Methods of chemical analysis of manganese ores	6·00
1493-1959	Methods of chemical analysis of iron ores	7·00
1527-1960	Methods of chemical analysis of fireclay and silica refractory materials	5·50
1546-1960	Methods of determination of arsenic in iron and steel	1·50
1559-1961	Methods of chemical analysis of ferro-alloys	10·00
1760-1962	Methods of chemical analysis of limestone, dolomite and allied materials	4·00
1917-1962	Methods of chemical analysis of quartzite and high silica sand	4·50
1940-1961	Methods of chemical analysis of tin ingot	3·50
1952-1963	Methods of chemical analysis of nickel anodes	5·00
1953-1962	Methods of chemical analysis of silver anodes	1·00
2000-1962	Methods of chemical analysis of bauxite	4·50
2017-1967	Methods of chemical analysis of metallic manganese	7·00
2018-1964	Methods of chemical analysis of calcium silicon	2·00
2277-1964	Methods of chemical analysis of metallic silicon	4·00
2411-1963	Methods of chemical analysis of flourspar (fluorite)	4·00
2599-1963	Methods of spectographic analysis of high purity zinc and zinc base alloys for die casting	4·50
2600-1964	Methods of chemical analysis of high purity zinc and zinc base alloys for die casting	3·50
3186-1965	Methods of chemical analysis of cadmium copper	1·50
3187-1965	Methods of chemical analysis of copper-nickel-zinc alloys	3·50
3200-1965	Methods of chemical analysis of cryolite	3·50
3685-1966	Methods of chemical analysis of brasses	5·00
3863-1966	Methods of chemical analysis of copper-tellurium alloys	1·50
4027-1967	Methods of chemical analysis of bronzes	8·00
4104-1967	Methods of chemical analysis of rutile	5·50
4129-1967	Methods of chemical analysis of aluminium trifluoride	5·00

PUBLICATIONS OF INDIAN STANDARDS INSTITUTION

INDIAN STANDARDS

About 4 500 Indian Standards, broadly classified under the following main heads, have been issued so far:

- Agriculture & Food
- Chemical
- Civil Engineering
- Consumer Products
- Electrotechnical
- Mechanical Engineering
- Structural & Metals
- Textile

Of these, the standards belonging to the Structural & Metals Group fall under the following categories:

- Copper and Copper Alloys
- Cranes and Allied Appliances
- Design Codes
- Ferro-alloys
- Foundry
- Lead, Zinc, Tin, Antimony and Their Alloys
- Light Metals and Their Alloys
- Metallic Finishes
- Ores and Raw Materials
- Pig Iron, Cast Iron and Malleable Cast Iron
- Precious Metals
- Refractories
- Solders
- Steel Castings
- Steel Forgings
- Steel Products, Wrought
- Steel Tubes and Pipes
- Structural Engineering Handbooks
- Structural Shapes
- Welding
- Unclassified

OTHER PUBLICATIONS

	Rs
ISI Bulletin (Published Every Month)	
Single Copy	2·00
Annual Subscription	15·00
Annual Reports (from 1948-49 onwards)	2·00 to 3·00 each
Handbook of ISI Publications, 1967	5·00

Available from

INDIAN STANDARDS INSTITUTION

Headquarters

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 1

Telephones 27 36 11 - 20 Telegrams Manaksanstha

Branch Offices

Telegrams Manaksanstha

534 Sardar Vallabhbhai Patel Road	Bombay 7	Telephone	35 69 44
5 Chowringhee Approach	Calcutta 13	„	23- 18 23
Industrial Estate Administrative Building, Sanatnagar	Hyderabad 18	„	3 92 29
117/418 B Sarvodaya Nagar	Kanpur	„	8 76 95
54 General Patters Road	Madras 2	„	8 72 78

Printed at Standard Printing Works, New Delhi 1, India